Gelcasting and thixotropic casting of industrial ceramics

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Abstract

Ceramic components made from aluminium titanate are in high demand due to the materials desirable mechanical properties. Many aluminium titanate components are currently manufactured by slip casting and isostatic pressing. However, these manufacturing techniques place limits on the size and geometrical complexity that is possible. Gelcasting and thixotropic casting are two novel ceramic forming processes capable of producing complex, solid aluminium titanate components which cannot be produced by current production methods. This paper describes the development of both thixotropic and gelcasting process for aluminium titanate.

1.0 Introduction

Rojan Advanced Ceramics is an industrial ceramics company which produce high value added ceramic components into a range of different industries. Recently Rojan has begun producing aluminium titanate components via slip casting and cold isostatic pressing. However, these manufacturing techniques place limits to the size and geometrical complexity that is possible. Hence, this project is aimed at overcoming such constraints by develop two related manufacturing processes to produce sintered aluminium titanate components, namely thixotropic (vibro)casting and gelcasting. These two novel techniques will allow Rojan to produce solid, complex components such as degassing impellers by gelcasting and kiln chutes by thixotropic casting to establish themselves in the niche market for specialized, value added products in the aluminium casting industry.

2.0 Background

2.1 Gelcasting

2.1.1 Introduction

Gelcasting was first developed in 1985 by scientists from Oak Ridge National Laboratory, USA. The gelcasting process is a novel ceramic forming process which can be used to produce solid, complex ceramic parts with excellent density, strength and form properties and which are unable to be replicated by other commercial ceramic forming processes. Gelcasting involves the addition of a small quantity of bio-polymer to a low viscosity, high volume fraction, ceramic suspension which is then poured into a mould. An initiator is then introduced to the suspension which causes it to gel. The gelled body has sufficient mechanical strength to be de-moulded and handled before traditional drying and sintering methods are used to produce the final part. Along with the ceramic powder, gelcasting requires a gelling agent, a dispersant and a small quantity of water.

2.1.2 Ingredients

Successful gelcasting requires a dispersant, a gelling agent, a small quantity of water, an initiator and the ceramic powder to be cast.

The dispersant is required to aid in mixing by causing an electrochemical process to prevent agglomeration. There is a standard dose of dispersant depending on the particle size of the ceramic powder. This amount of dispersant will assure that with enough mixing the whole proportion of particles are electrostatically coated and the mixture is fully dispersed.

The gelling agent is required to gel the slurry so it will hold its shape once the mould is removed, and then during traditional drying and sintering cycles. Gelatine has been chosen as the gelling agent for this project because it is cheap, readily available and is not harmful. The concentration of gelatine in water must be greater than 3%wt relative to water, to allow solid formation and less than 3.5%wt to allow fluid slurry [2].

A small quantity of water is required to allow the slurry to flow and to dissolve the gelatine and form a gel. Increasing the proportion of water to ceramic powder will reduce the viscosity of the slurry, and will also reduce the density of the resulting gelled green body. A higher green state density will result in less shrinkage during sintering.

A temperature increase will initiate the gelling process. Once the heated slurry is cast, placing the mould in a refrigerator over night is sufficient to form a gel.

2.2 Thixotropic

2.2.1 Introduction

Thixotropic (or vibro) casting is a little-known derivative of solid slip casting, and was first reported as being used in the refractories industry in the early 1970's. This kind of casting technology has also been used extensively in the cement industry. Thixotropic casting involves specific particle size combinations which, when mixed with water and a small quantity of binder, produces a slurry which flows easily under vibration. The slurry is poured into a vibrating mould and sets once the mould ceases to vibrate. The part is left to dry, extracted then sintered.

2.2.2 Ingredients

Particle packing is the key to successful thixotropic casting. Combining the correct proportions of specific particle sizes will create sufficiently dense slurry, when mixed with the remaining ingredients. Typical particle sizes range from a few millimetres to sub micron sized powder.

A small quantity of binder is required to maintain the shape of the cast in its green state. The binder used for thixotropic casting is a special type of clay produced for refractory use.

A dispersant is added to the mixture to prevent agglomeration. The dispersant used in thixotropic casting for this project was different to that added to the gelcast slurry but the mechanism is the same.

A small amount of water is added to the mixture to produce thick slurry which will only flow under vibration. The amount of water required is related to the aggregate surface area of the particles in the mixture. A greater proportion of fine particles in the mixture will require a greater quantity of water.

A quantity of sugar (<1%wt) can be added to attain a white surface finish. This small amount of sugar will not affect any other properties of the slurry or the sintered component.

3.0 Experimental

3.1 Gelcasting

Gelcast recipe (per 500g of ceramic powder):

- 5grams GELATB70
- 145grams water
- 5grams dispersant [Dispex]

3.2 Thixotropic casting

Powders:

• 3.35-2mm	27%
• 2-0.5mm	21%
• 0.5-0.2mm	6%
• <0.2mm	16%
• <63_mm	25%
• Clay Ceram	5%

Other:

• Dispersant [Flintox]	2.7%wt/wt of powder
• Water	11%wt/wt of powder
• Sugar	<1%wt/wt of powder

4.0 Casting procedure

4.1 Gelcasting

The procedure for gelcasting begins with combining the ingredients and mixing them thoroughly by hand, and then for at least one hour in a ball mill. Once the slurry is mixed it must be heated and degassed before it can be cast in a mould.

4.1.1 Mixing

Powders and liquids are mixed separately then combined before being placed in a ball mill. Milling media used in ball mills are generally ceramic balls with a diameter of a couple of centimetres. The quantity of milling media should be about equal to the quantity of slurry for effective mixing.

4.1.2 Heating

Heating the slurry will reduce its viscosity. Heating the slurry filled container straight from the ball mill will allow the milling media to be removed easily and will also aid in degassing. Heating the slurry to 60°C for 30 minutes is sufficient to dissolve the gelatine.

4.1.3 Degassing

Degassing is performed under vacuum to remove air trapped in the slurry during milling. Degassing is crucial to achieving a dense, defect free part. The heated slurry is placed in a vacuum and agitated to break up any surface skin. The slurry remains under vacuum for about 15 minutes until the slurry settles down to a slow boil and the degassing process is complete.

4.1.4 Casting

Once the slurry has been degassed, re-heating slightly will allow the slurry to flow easily into the mould. The mould and any utensils used must be heated along side the slurry prior to casting, because cold surfaces will cause the slurry to thicken upon contact. Some typical mould materials are aluminium, PVC or plaster coated in a waterproof sealant. The slurry will flow more freely and any trapped air pockets are purged if casting is done on a vibrating table.

4.2 Thixotropic casting

4.2.1 Mixing

Powders and liquids are mixed separately then combined to form thick, abrasive slurry. Mixing for thixotropic casting is performed in a mixer suitable for mixing concrete, or by hand for small quantities.

4.2.2 Casting

Casting is performed under vibration to allow the slurry to flow into the mould. Moulds are made from plaster with heavy sections re-enforced with steel mesh and bars. The moulds are strapped to the vibrating table and the slurry is poured into the cavity from above. Once the cavity is filled, the mould remains on the vibrating table for a minute or two to work out any trapped air pockets.

5.0 Drying

5.1 Gelcasting

Once the slurry has formed a gel, it must be de-moulded and placed on a mesh tray in a controlled humidity environment. Conditions inside a refrigerator are acceptable for controlled humidity drying. Drying times vary according to the thickness of parts, but for thicknesses of about 1-2 centimetres, 4 or 5 days in the refrigerator is adequate. Once parts have dried solid in the refrigerator, they are moved to an oven at temperatures up to 75°C for about 12 hours to complete the drying process. Thin, flat components may require small weights placed on all edges to prevent any bowing during drying.

5.2 Thixotropic casting

The slurry begins to dry as soon as it is poured into the mould, due to water being drawn out by capillary action at the mould surface. The plaster mould continues to draw water from the slurry until it has solidified enough to support its own weight, at which time it is de-moulded. De-moulded parts are left to air dry for at least 4 days once they have been de-moulded, again depending on part thickness. Before sintering, parts are completely dried in an oven at temperatures up to 100 C for ~24 hours.

6.0 Sintering

6.1 Gelcasting

6.1.1 Dilatometry

Gelcast aluminium titanate components can be sintered at temperatures up to 1700°C. The dilatometry plot below shows the %shrinkage as the sample is heated to 1400°C at a constant rate.

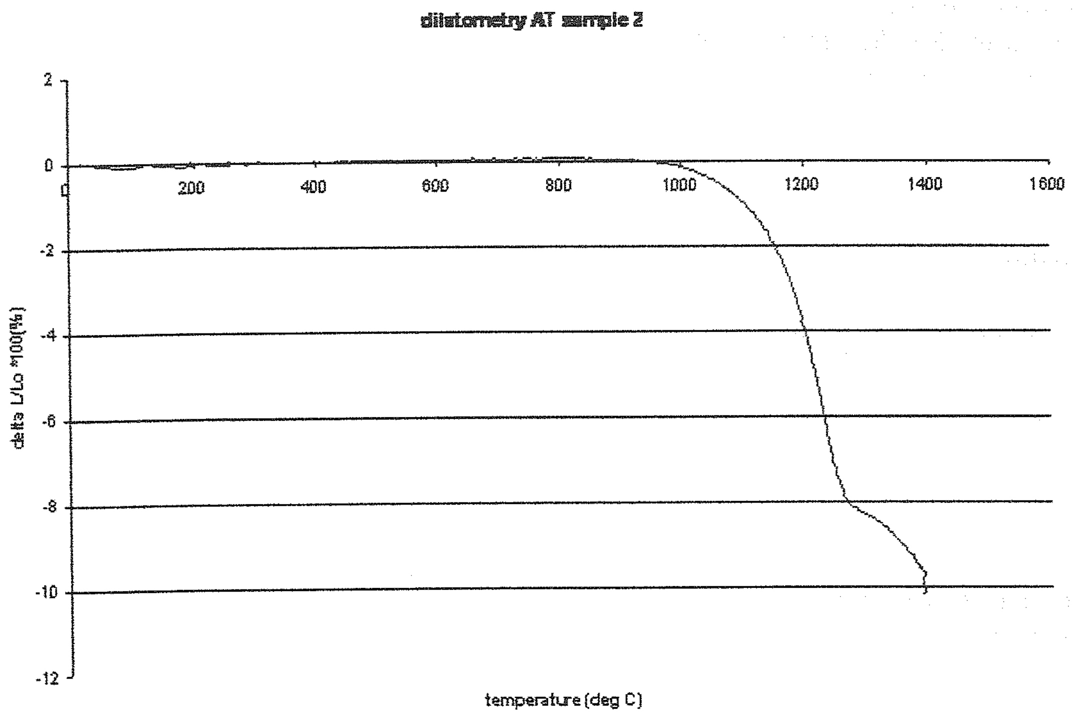

Figure 1: Dilatometry results for gelcast aluminium titanate [% shrinkage vs temperature]

6.1.2 Bulk density results

The chart below shows the bulk density of five sets of three samples, each set sintered to a specific peak temperature. The density measurements below were conducted using the immersion method; these results are confirmed by geometrical density measurements. The theoretical density of aluminium titanate is 3.7grams/cm^3.

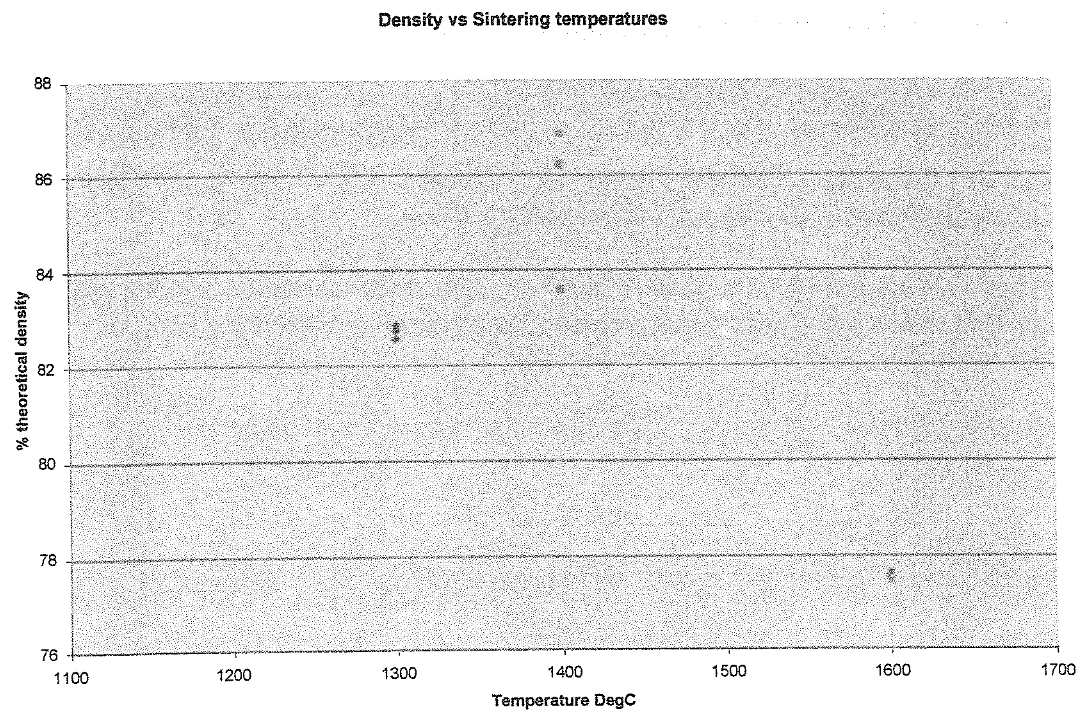

Figure 2: Bulk density results for sintered, gelcast aluminium titanate samples [g/cm^3].

6.2 Thixotropic casting

The sintering program for thixotropic cast aluminium titanate components is the same as that used for gelcasting because they are both essentially the same material, although, the gelcast parts are made from virgin aluminium titanate and the thixotropic cast parts are made from pre-sintered material. The bulk density results for the thixotropic cast samples are not yet available.

7.0 Impending work

7.1 Gelcasting

To complete the research on the sintering aspects of gelcasting, the sintered test samples must be mounted and polished so their microstructure can be optically analysed. After studying the microstructure of the samples which have been sintered to different temperatures, the best microstructure will indicate the most appropriate temperature to sinter gelcast aluminium titanate components.

7.2 Thixotropic casting

Density measurements still remain to be completed on the test samples, to determine the best sintering temperature, in terms of maximum density. Similarly to the gelcast impending work, the thixotropic cast samples must also be mounted and polished to assess their microstructure and how it relates to peak sintering temperature.

8.0 References

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